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THE EFFECT OF PRELIMINARY RECOVERY ON THE RECRYSTALLIZATION KINETICS OF DEFORMED METALS

L. N. Larikov, O. E. Zasimchuk, and Zh. Ya. Kutikhina

Questions pertaining to the effect of previous annealings at low temperatures on the course of the recrystallization process are disputable (see for example (1-5)). In this report are given such investigations on nickel, copper, niobium, molybdenum, tungsten monocrystals, and also on polycrystalline copper and nickel. Using copper of type BK (99.98% Cu), electrolytical nickel (99.99% Ni), purified by electron-radiation remelting in vacuum, and Nb, Mo, W, monocrystals, grown with the aid of electron ray.

The copper monocrystal, oriented along the axis (013), which had the form of a cylinder 8 cm long, diameter of 0.34 cm, has weakly deformed by bending around a crystallographic direction (121). Since the axes of bending and sample formed an angle, not equalling 90°, the bending of the crystal was carried out by a spiral with internal diameter of 9.3 mm. After deformation the crystal was cut into sections, which appeared to be weakly bent cylinders of 3-5 cm in length. At a small rise on the surfaces of the samples appeared two systems of sliding planes, which is a necessary condition for the recrystallization during heating (6).

Annealing recrystallizations were carried out at a temperature of 900°C within one hour, and the preliminary ones -- at a temperature range of 500–750° within various time intervals. The presence of recrystallization plots were confirmed by new point reflexes on lauerograms.

The monocrystal, oriented along the axis 111, was rolled in plane 110 in direction 112 by ~80°. For rolling in the given plane and to assure a constant degree of deformation in length the crystal was ground from two sides. After the rolling the crystal was cut into sections with a dimension of 1 x 10 x 3 mm.

Recrystallization and preliminary annealing of the rolled monocrystal were carried out at a temperature range of 100–170°C at a strict adherence of isothermal conditions (7). The appearance in the volume of the sample of recrystallization centers of a visible dimension (of the order of 10^{-4} cm) appeared microstructurally (8).

The obtained results are given in table. From the table is evident, that preliminary annealings of a highly deformed monocrystal does not affect the recrystallization process. Preliminary recoveries of a bent monocrystal within various time periods and at various ignition temperatures, lead however, to recrystallization delays at the subsequent high temperature annealing (800°C).

An analogous effect of recrystallization delay by preliminary recoveries were observed by Zemel and Makhlin (4) when studying a weakly deformed by bending silver monocrystal; this effect binding same with the yield of excessive vacancies during preliminary annealing. Total dislocation density, revealed by the microstructure by the etching pits, remained in them unchanged.

However, as shown by data of many authors (e.g. (9)) the output of excessive vacancies in deformed polycrystalline copper takes place at low temperatures, close to room temperature. It was therefore of interest to compile the results, obtained during the study of copper monocrystals, very much and weakly deformed, with the results of polycrystalline investigation.

Table

State of sample deformation	Method	Preliminary annealing		Recrystallization annealing		Res.
		t°, C	hour	t°, C	hour	
Cu, monocrystal; weak deformation by bending	x-ray	–	–			+
		750	2 hrs			–
		–	–			+
		680	1 hr 30 min			–
		–	–			+
		670	37 hrs			–
		–	–	900	1 hr	+
		560	1 hr 40 min			–
		–	–			+
		500	67 hrs			–
		–	–			+
		470	1 hr 40 min			–
Cu, monocrystal; rolling $\varepsilon \sim 85\%$	x-ray	–	–			+
		50	1 hr			+
		–	–			+
		100	1 hr	170	1 hr	+
		–	–			+
		150	1 hr			+
Cu, $\varepsilon \sim 3\%$		–	–			+
		320	1 hr			–
$\varepsilon \sim$ [illegible]%		–	–			+
		320	1 hr	425	1 hr	+
polycrystal, rolling	micro-structural					
$\varepsilon \sim 5\%$		–	–			+
		320	1 hr			+
$\varepsilon \sim 7\%$		–	–			+
		320	1 hr			+
Cu, polycrystal compr $\varepsilon \sim 80\%$ t° def 20°C	x-ray	–	–			+
		20	960 hrs			+
		–	–			+
		20	8700 hrs			+
		–	–	167	1 hr	+
		50	1 hr			+
		–	–			+
		98	1 hr			+

(table continued on next page)

(table continued from previous page)

Cu, polycrystal compres. $\varepsilon \sim 80\%$ t^{o} def 183°C	x-ray	– 100 – 100 – 20	– 30 min – 10 min – 24 hrs	155	1 hr	+ + + + + +
Ni, monocrystal compres. $\varepsilon \sim 4\%$	micro-structural	– 400	– 1 hr	600	1 hr	+ –
Ni, polycrystal compres. $\varepsilon \sim 80\%$	x-ray	– 20 – 20 – 100 – 200	– 960 hrs – 8760 hrs – 1 hr – 1 hr	320	1 hr	+ + + + + + + +

The plus sign marks samples, which were recrystallized at the mentioned annealings, minus sign marks samples which have not recrystallized as a result of preliminary recovery.

Samples of polycrystalline copper were annealed at a temperature of 600°C (10-15 min) for removal of the cold hardening after mechanical processing and they deformed by monoaxial depressions by ~80% (highly deformed polycrystals also by rolling by 3-7% (weakly deformed polycrystals). Preliminary processing of the latter led to sharp plastic deformation (90%) and recrystallization annealing at 400°C. Such a processing assured the obtainment of a thin foil (0.004 cm) from steel of fine grain (of the order of foil thickness) (10). The recrystallization centers were revealed on the background of the matrix, when their dimensions exceeded the maximum dimension of grains of the matrix (2). Recrystallization annealings of weakly deformed polycrystallines were carried out at a temperature of 425 ± 2°C, previously at 320 ± 2°C within 1 hr. Deformation was carried out ~80° at room temperature and at a temperature – 183°. Temperatures and hours of previous and recrystallization annealings are given in table.

As is evident from the table, in highly deformed polycrystals and monocrystals of copper previous recoveries do not affect the recrystallization process In case of weakly deformed polycrystallines is sometimes observed the effect of recrystallization delay by preliminary recovery. It should be mentioned, that during ~3% deformation in the samples, which have not become recrystallized as a result of preliminary return, certain grains had a clearly expressed substructure (Fig. 1), has been revealed after the annealing. This allows to assume, that in the given case during the annealing took place polygonization (11).

To an analogous conclusion leads also the structural analysis of spots on the lauerograms of the bent monocrystals, which have not become recrystallized as a result of preliminary recovery. The washed out spots of the deformed crystal split (Fig. 2), which is characteristic of the material, which experiences polygonization (12).

In this way, the recrystallization delay because of preliminary recovery is observed only in weakly deformed mono- and polycrystals, whereby in polycrystals at very low deformations. Maybe, this is bound with a different dependence of polygonization and recrystallization rate upon the degree of deformation (11).

It should be mentioned, that polygonization in copper to be observed, is very important (13). This is bound with low energy of packing defects in copper in comparison with other face centered cubic metals (14).

The effect of recrystallization delay of preliminary polygonization observed by us also on a nickel monocrystal, deformed by 4% in plane (111). In highly deformed (80%) nikel preliminary recovery does not affect the recrystallization process (see table).

Preliminary recovery delays and even fully prevents recrystallization only at very low deformations of mono- and polycrystals of copper and nickel. At considerable deformations (~80%) preliminary recovery does not affect the recrystallization process.

Fig. 1. Grain structure of polycrystalline copper sample, deformed by rolling by 3% which has not become recrystallized at 425°C (1 hr) as a result of preliminary annealing at 320°C (1 hr). (Arrows show the subboundaries in the center of the grain, x500).

Delay in recrystallization by preliminary annealing is bound with polygonization, whereby at small deformations of the monocrystal the condition of polygonization is fully weakened, and recrystallization does not take place. At somewhat greater degrees of deformation preliminary polygonization leads only to a delay in recrystallization at successive heating.

In case of polycrystallines, where during deformation takes place harmonious interaction of other crystals, the removal of recrystallization by polygonization can still be observed at lesser deformations, than in monocrystals (11).

Removals of recrystallization, thanks to polygonization is observed by us also at annealings of deformed monocrystals of high melting metals: niobium, tungsten, and molybdenum (Fig. 3). When they are deformed by rolling to 30% in plane (110) the weakened during the heating are bound only with the polygonization process. An analogous result was obtained by us when investigating bent monocrystals.

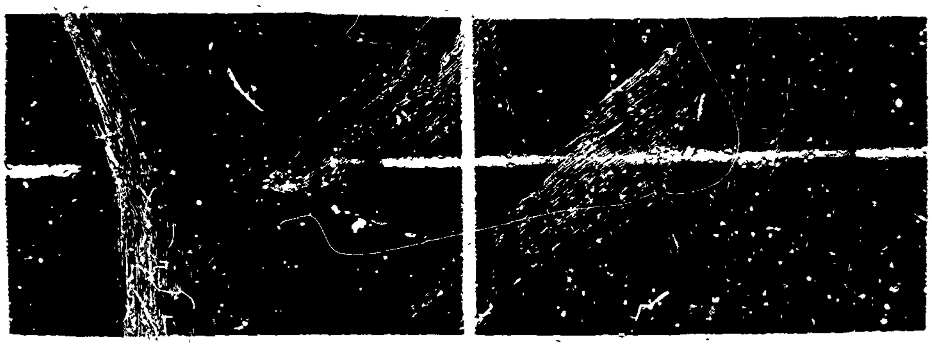

Fig. 2. Structure of Flames or Lauegrams of a Deformed Copper Monocrystals.

KEY: (a) after bending; (b) after burning at 500°C for 67 hrs and at 900°C for 1 hr.

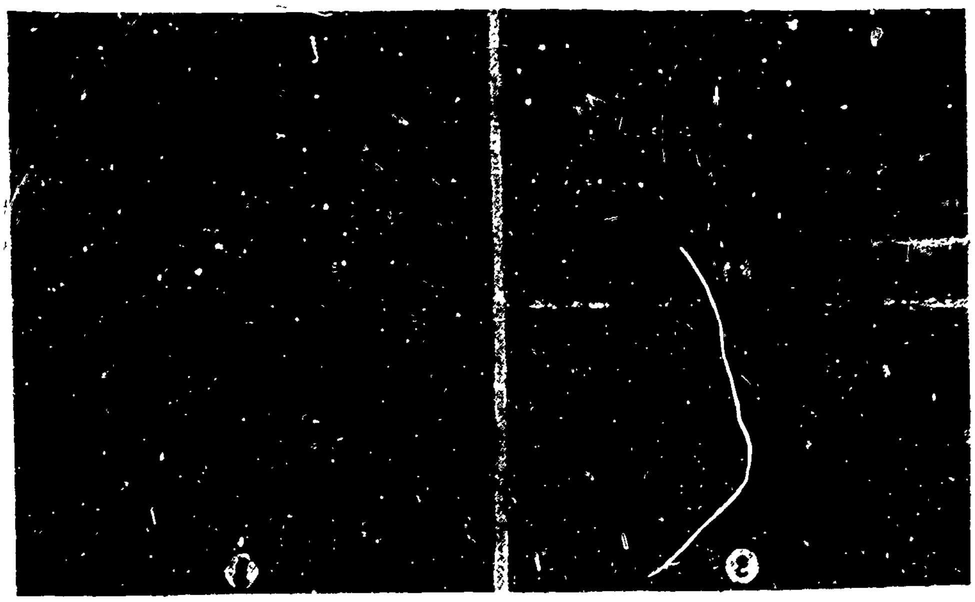

Fig. 3. Structure of spots on lauegrams of deformed monocrystallites, which have not recrystallized as a result of polygonization.

KEY: (a) molybdenum, deformed by bending; (b) the very same sample, but annealed within a period of 1 hr at 2200oC; (c) molybdenum, rolled by 15% in plane (110); (d) the very same sample, annealed within 1 hr at 2300°C.

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Summary

X-ray and microstructural methods were used to study the effect of preliminary annealing on subsequent recrystallization in single crystals of a number of pure metals (Cu, Ni, Nb, W, Mo).

After weak deformations preliminary recovery of the samples leads to a delay and, sometimes, even to complete prevention of subsequent recrystallization. Single crystals of metals with body-centred lattices may be deformed by 30% and more without recrystallization during subsequent annealing, provided there is a definite geometry of plastic deformation.

The effect of inhibition of recrystallization by preliminary annealing was observed earlier by Semell and Machlin [4] on studying a weakly bent silver single crystal, and they connected it with the vanishing of excess vacancies during preliminary annealing. It is shown in this paper that in the investigated metals preliminary annealing inhibits subsequent recrystallization owing to polygonization rather than to vacancy rest.

After strong (80%) deformations preliminary polygonization did not affect the subsequent course of recrystallization.